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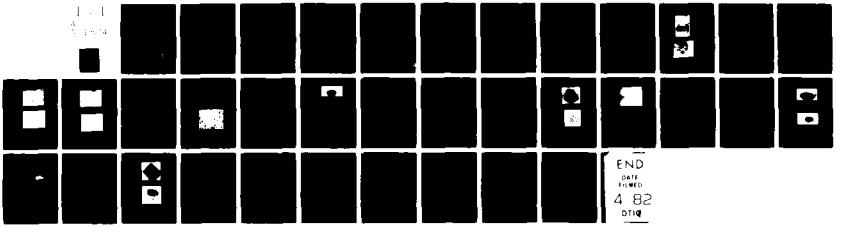

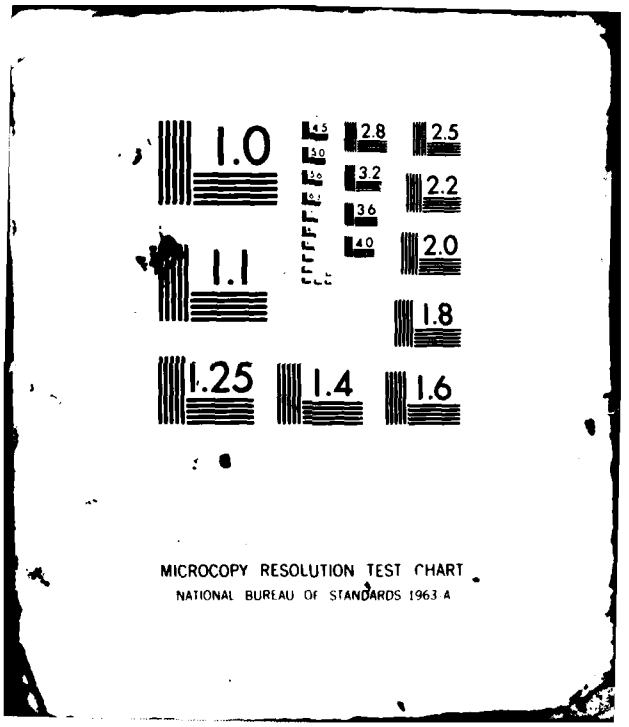
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Report N00014-80-C-0437-1

# STUDY OF MECHANO-CHEMICAL MACHINING OF CERAMICS AND THE EFFECT ON THIN FILM BEHAVIOR

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June 1981

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(U) Efforts were made to finish the surfaces of single-crystal MgO, Si, and both single-crystal and tape-cast $Al_2O_3$ using the technique of mechano-chemical polishing. In this technique, the emphasis is on employing an abrasive whose hardness is less than that of the workpiece to produce damage-and scratch-free surfaces that are also flat. An evidence for mechano-chemical polishing in the form of scratch-free surfaces was not obtained in the case of MgO, which was polished using talc, rock salt, calcite, and fluorite as abrasives. The mechano-chemical polishing effects, however, were readily observed when Si was polished using $CaCO_3$, $BaCO_3$, and MgO as abrasives, and when $Al_2O_3$ was polished on a plate of window glass using no abrasive.

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20 . Abstract - Mechano-chemically-polished surfaces were characterized using RHEED, profilometry, interferometry, optical microscopy, and electron spectroscopy for chemical analysis.

# Preface

This report covers work performed under Office of Naval Research Contract No. N00014-80-C-0437 during the period 1 May 1980 to 30 April 1981. The authors would like to thank Dr. K.D. McHenry for his association with this project in its early stages, Ms. J.M. Hanson for experimental measurements, and Mr. D.J. Sauve, Jr. for excellent technical support throughout this work. ESCA data of mechano-chemically-polished samples were obtained by Dr. T.W. Orent and Mr. A.D. Beck, and RHEED and X-ray work was performed by Mr. C. Knudson.





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# Table of Contents

## List of Illustrations



## List of Tables

# Section 1
# Introduction

In a variety of applications, particularly those associated with optics and electronics, materials possessing smooth, damage-free, and flat surfaces are required. Such surfaces are difficult to obtain using conventional techniques in which abrasives with hardnesses greater than that of the workpiece are employed. Hard abrasives introduce scratches and a damaged layer, which must be removed by a final treatment. This treatment often consists of chemical polishing in which the material is removed by chemical rather than mechanical means. However, chemical polishing rapidly degrades the flatness of the workpiece and often introduces orange-peel structure on the surface. In order to overcome these problems, the technique of mechano-chemical polishing was recently introduced by Yasunaga et al.,[1] who have reported that this technique can yield damage- and scratch-free flat surfaces at surprisingly high rates of material removal.

In mechano-chemical polishing, the emphasis is on employing an abrasive whose hardness is less than that of the workpiece. Since such an abrasive cannot damage or scratch the workpiece, the technique yields damage- and scratch-free surfaces. Although the abrasive employed in mechano-chemical polishing is soft, it yields high removal rates because of the chemical reaction that occurs at the contact points of the abrasive and the workpiece. The transformed regions are believed to be extremely small (of the order of 100Å), and the resulting phase (since its properties differ from those of the workpiece and abrasive) is removed by subsequent contact with other abrasive grains.

Mechano-chemical polishing is performed in a manner similar to conventional polishing using conventional polishing machines. However, in order to promote a chemical reaction, it is often necessary to generate high temperatures at the contact points of the abrasive and the workpiece. A simple way to accomplish this is by applying high pressure on the workpiece and performing the mechano-chemical polishing dry. In some cases, chemical reactions can be induced at moderate temperatures, and significant removal rates are observed when the mechano-chemical polishing is performed wet. The term wet refers to employment of a polishing medium consisting of a slurry of soft abrasive in water, as opposed to use of loose powder in dry polishing. In any case, one expects the applications of mechano-chemical polishing technique to be restriced to hard materials, that is, those materials which can withstand pressure required to react chemically with the abrasive without deformation or fracture.

The work performed during this contract had two long-term objectives: 1) to understand the mechanisms involved in mechano-chemical finishing by better characterization of

1. N. Yasunaga, N. Tarumi, A. Obara, and O. Imanaka in The Science of Ceramic Machining and Surface Finishing II, B.J. Hockey and R.W. Rice, editors, National Bureau of Standards Special Publication 562, 1979, p. 171.

the topography and chemical strucure of both the finished surfaces and the residual polishing powders, and 2) to determine the consequences of mechano-chemically-finished surfaces on the physical performance of materials.

Physical properties of interest in this program were mechanical strength, and electrical conductivity and adhesion of metal films. Mechanical strength is a direct physical property in that it is directly affected by the surface conditions, whereas electrical conductivity, or adhesion of metal films, is an indirect physical property, which is influenced by the surface finish of the substrates.

Efforts have been made to mechano-chemically polish three different materials, which, in the increasing order of hardness are single-crystal magnesia, single-crystal silicon, and single-crystal and tape-cast alumina. No evidence for mechano-chemical polishing was obtained in the case of magnesia. However, the contractor was able to confirm the occurrence of mechano-chemical polishing effect in the case of silicon, as well as alumina. This report describes the results of the contractor's efforts to mechano-chemically polish these materials, as well as to characterize the mechano-chemically-polished surfaces, using several analytical techniques.

# Section 2
# Experimental

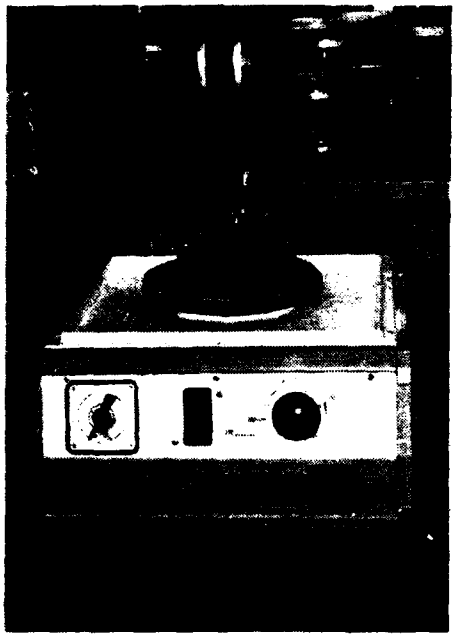
Attempts to mechano-chemically polish various materials were made on two polishing machines: a Precision Polishing Machine Model PM2, manufactured by Hacker Instruments, Inc. of Fairfield, New Jersey, and a Strasbaugh Polishing Machine, Model 6Y-1, manufactured by R. Howard Strasbaugh, Inc. of Huntington Beach, California. The machines are shown in Figure 1. In the tests conducted on the Precision Polishing Machine, the pressure acting on the sample was readily determined by measuring the weight of the sample holder, adding to it any extra weights placed on the sample holder, and dividing the total with the surface area being polished.

The Strasbaugh Polishing Machine used in the tests was equipped with an air pressure system, the control unit of which contained a gauge which showed pressure in psi. This pressure could be adjusted over a range from 0 to 100 psi. In a typical experimental situation, 1 psi translated into a load on the sample of approximately 1 lb (0.45 kg).

Mechano-chemical polishing experiments were conducted both in wet and dry conditions. In the dry condition, the lap was simply covered with the desired abrasive at the start of the run and replenished as required. In the wet condition, a slurry containing 2-10 weight percent of soft abrasive was sprayed on the lap frequently to ensure that the wet condition remained throughout the experiments.

Attempts to measure removal rates were made for MgO, $Al_2O_3$, and Si. MgO single crystals used for polishing studies were of dimensions 0.75cm × 0.75cm × 0.5-1mm. All six faces of these crystals were cleaved along {100} planes. The procedure used for computation of the removal rates of MgO was to accurately measure the cross-sectional area of the sample and its thickness before and after polishing for a given period of time. From these data and the known density of MgO (4.0gm/cc), the removal rates in mg/hr were computed. Removal rates in the case of $Al_2O_3$ were determined by measuring the decrease in size of Knoop indentations made using a load of 300 grams. It was necessary to make a series of indentations on a given specimen because only 2 or 3 out of 10 indentations retained their shapes during polishing; several cracks appeared near the remaining indents and accurate measurements of their size as a function of polishing time could not be made. The Knoop indentation technique was tried, with little success, to determine the wear rates of silicon single crystals.

Some caution should be exercised in connection with the removal rate data. There are many factors that contribute to the removal rates obtained in mechano-chemical polishing, some of which are known and can be controlled readily (e.g., load on the sample and the polishing wheel speed). There are others which influence removal rates in a manner that is not clearly understood at present and attempts to control them have not been made yet. Some examples of these factors are the initial flatness and surface roughness of the sample, how the lap is charged with the abrasive, whether the lap has been used previously or not, and in the case of wet mechano-chemical polishing of silicon single crystals, the concentration and pH of the slurry.



(a) Precision Polishing Machine, Model PM2 (Hacker Instruments)

(b) Strasbaugh Polishing Machine, Model 6Y-1

Figure 1 Photographs of polishing machines

# Section 3
# Results

## MAGNESIA

Single crystals of magnesia were selected for the present study because they could be cleaved easily along {100} planes. In addition, the dislocations in MgO can be revealed easily by the etching techniques, a fact that has contributed significantly to the understanding of the deformation and fracture behavior of this material.

Attempts to mechano-chemically polish MgO single crystals were made on the Precision Polishing Machine using talc ($3\ MgO \cdot 4SiO_2 \cdot H_2O$), rock salt (NaCl) calcite ($CaCO_3$) and fluorite ($CaF_2$) as soft abrasives. The respective hardnesses of these abrasives on the Mohs scale are 1, 2, 3, and 4, as compared to ~5 for magnesia. Some magnesia crystals were polished using these abrasives on a smooth glass lap. Since most glasses are harder than magnesia, they can scratch magnesia, and thus, contribute to the observed removal rates. For this reason, several MgO crystals were polished on a brass wheel covered with an adhesive-backed Nylon cloth*. The laps of both types were ~25cm in diameter.

In mechano-chemical polishing, the load applied to the sample during polishing and the polishing speed are two important parameters. In the case of MgO crystals of 0.75cm × 0.75cm in cross section, the applied load was either 1.36kgs or 4.4kgs. At higher loads, MgO crystals fractured frequently and the polishing experiments could not be conducted successfully. The rotation speed of polishing laps was either 30 r/min or 60 r/min during polishing of MgO.

Following polishing, samples were examined in an optical microscope to see if their surfaces were scratch-free. The samples were then etched in a solution of 5 parts saturated ammonium chloride ($NH_4Cl$), 1 part concentrated sulfuric acid ($H_2SO_4$), and 1 part distilled water, and examined again. Some polished MgO crystals were cleaved on a plane perpendicular to the polished surface and etched in the above solution to determine the extent of sub-surface damage.

Effects of various polishing parameters on the removal rates of MgO are summarized in Table 1. It is seen that a material removal took place in every polishing situation attempted. It was suggested earlier that glass, since it is harder than MgO, could contribute to the observed removal rates when used as a lap material. The observed removal rates, however, were not consistently higher when MgO was polished on a glass lap, as compared to Nylon-covered brass lap. For example, the removal rates observed in attempts to wet mechano-chemically polish MgO using talc or $CaCO_3$ were higher when polished on a Nylon covered brass lap.

* Buehler Ltd., Evanston, Illinois

TABLE 1. REMOVAL RATES (mg/hr) FOR MAGNESIUM OXIDE SINGLE CRYSTALS

| | | | | Polishing Environment | | | |
|---|---|---|---|---|---|---|---|
| | | | | Wet | | Dry | |
| | | | | Wheel Speed | | Wheel Speed | |
| | | | | Low (30 r/min) | High (60 r/min) | Low (30 r/min) | High (60 r/min) |
| Surface | Glass | Load | High | Talc 1.135<br>NaCl 5.499<br>$CaCO_3$ 3.719 | Talc 7.008<br>NaCl 6.66 | Talc 66.314<br>NaCl 0.797<br>$CaCO_3$ 139.89 | Talc 163.09<br>NaCl 5.634 |
| | | | Low | Talc 5.30<br>NaCl 2.287 | Talc 1.03<br>NaCl 6.785 | Talc 13.408<br>NaCl 2.165<br>$CaCO_3$ 2.39 | Talc 67.946<br>NaCl 0.84<br>$CaCO_3$ 6.34 |
| Wheel | Nylon | Load | High | Talc 11.91<br>$CaCO_3$ 4.06 | Talc 14.153<br>$CaCO_3$ 7.02 | Talc 1.18<br>NaCl 84.18<br>$CaCO_3$ 2.4<br>$CaF_2$ 8.45 | Talc 3.175 |
| | | | Low | Talc 26.94<br>NaCl 5.71 | Talc 16.73 | Talc 9.45<br>NaCl 36.04 | Talc 5.46 |

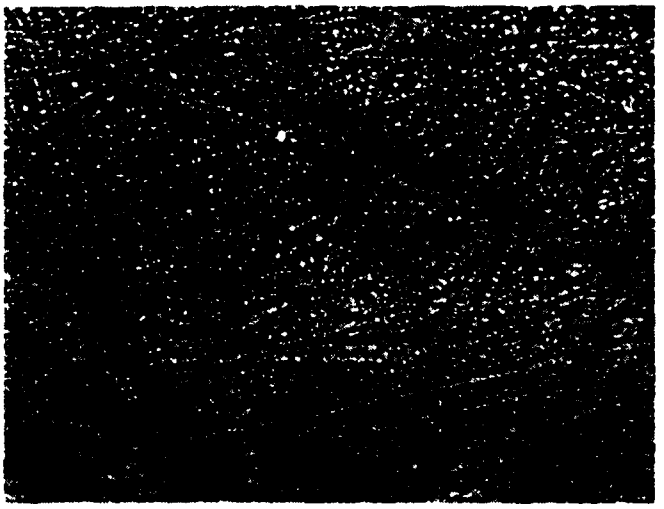
All powders screened to 325 mesh (45μm).

Metallographic examinations of the surfaces of polished MgO crystals gave no evidence of mechano-chemical polishing effect. The polished surfaces were scrached and damaged. Etching studies revealed a correlation between the removal rates and the extent of surface and sub-surface damage. High removal rates were associated with high dislocation density on the surface and, thus, greater extent of sub-surface damage (see Figure 2). Low removal rates were associated with smoother surfaces and a shallow sub-surface damage (see Figure 3).

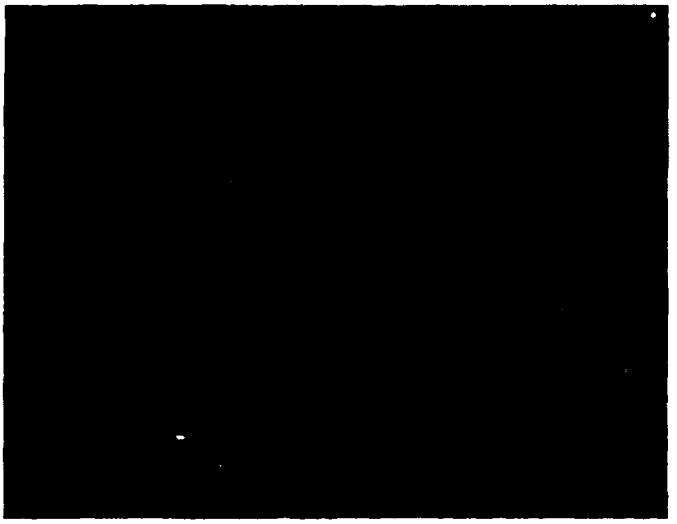
X-ray analysis of debris obtained by polishing MgO dry on glass using talc, and that obtained by polishing it wet on glass with NaCl gave diffraction patterns of only talc and NaCl. This data and the results of metallographic examinations indicate that true mechano-chemical effect did not occur in the case of MgO for the conditions chosen in this work. The material is apparently removed by plastic flow and mechanical impact fracture. Thus, magnesia is too plastic (or weak) to support high pressures and temperatures necessary at contact points in mechano-chemical polishing.

## SILICON

Most of the silicon single-crystal samples used in this work were 7.5cm in diameter, 0.05cm thick, and were of {100} orientation. Efforts to mechano-chemically polish these



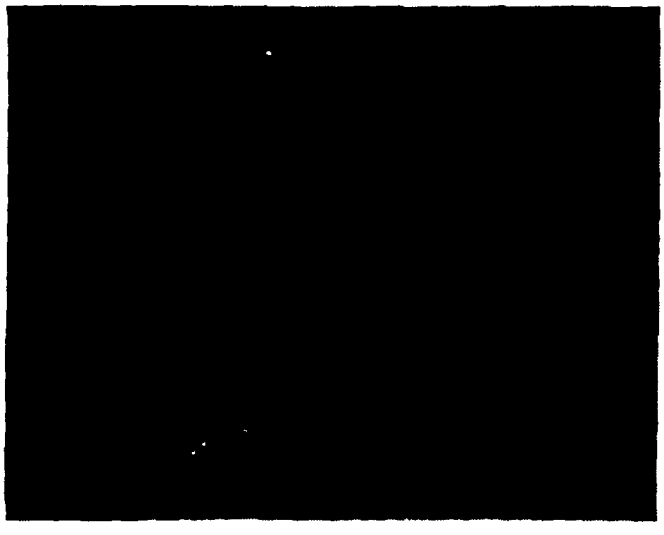
(a) Polished surface



(b) Cross section showing the depth of sub-surface damage

Figure 2. Photomicrographs of the etched surfaces of MgO polished dry on nylon using NaCl

(a) Polished surface



(b) Cross section showing the shallow depth of sub-surface damage

Figure 3. Photomicrographs of the etched surfaces of MgO polished wet on glass using NaCl

samples were made using three abrasives, $CaCO_3$, $BaCO_3$, and MgO, and using 25-cm diameter laps of either linen Bakellite or PVC. In a typical polishing attempt, a rotation speed of 50 r/min was employed for the lap and a pressure of about 100 gm/cm$^2$ was applied on the specimen. Under these experimental conditions, the laps of PVC wore out faster than the laps of linen Bakellite, and most of the polishing was performed on the laps of linen Bakellite.

Scratch-free and highly reflecting surfaces were readily obtained when silicon samples were wet or dry mechano-chemically polished using any of the above three abrasives. Slurries used for wet mechano-chemical polishing contained about 10 weight percent of the soft abrasives.

Attempts to measure the removal rates realized during mechano-chemical polishing of silicon using the Knoop indentation technique met with little success. Samples used for these measurements were 3mm in thickness and ~ 1cm × 1cm in cross section, and the Knoop indentations were made using the loads in the range 50-500g. Even at low loads, several cracks formed near the diagonals of the indents, resulting in a rapid distortion of their shapes during mechano-chemical polishing.

An indication of the rate at which silicon is removed during mechano-chemical polishing can be obtained from surface roughness data shown in Figures 4 and 5. Figure 4 shows the surface profile, as well as the Nomarski micrograph, of a 7.5-cm diameter silicon wafer before polishing. Figure 5 shows the surface profile and the Nomarski micrograph of the same silicon wafer after 2 hours of dry mechano-chemical polishing with $CaCO_3$. It can be seen that mechano-chemical polishing for 2 hours reduces the peak-to-peak roughness of silicon from a value of ~4$\mu$m to a value of ~40Å. This corresponds to a removal rate of at least 2$\mu$m/hr.

Figure 6 shows the reflection high-energy electron diffraction (RHEED) pattern of a sample of silicon which was wet mechano-chemically polished with $CaCO_3$. This pattern was obtained using a 100-kV electron beam which has a depth of penetration of ~50Å.[2] The presence of spots and the Kikuchi lines seen in the RHEED pattern indicates that the mechano-chemically-polished silicon surface is of high perfection.

Several samples of mechano-chemically-polished silicon were examined using the technique of electron spectroscopy for chemical analysis (ESCA). The objectives were to see if the polished surfaces were contaminated by the polishing abrasive or a reaction product of the polishing compound and the silicon, and to determine the extent of oxidation of silicon. The sensitivity of ESCA technique is about 0.001 monolayer, although it varies with material and experimental conditions. Of particular interest in the case of silicon is the $Si_{2P}$ peak (binding energy, Eb = 99eV) and the peak at Eb = 103eV which is caused by oxidation of silicon. The smaller the height of this peak relative to the $Si_{2P}$ peak, the smaller the extent of oxidation of silicon. Values reported for this ratio are 0.3 for conventionally-polished silicon samples and in the range of 0.12 - 0.18 for silicon samples polished mechano-chemically with $BaCO_3$.[1]

2. J.L. Whitton, J. Appl. Phys. 36, 3917 (1965).

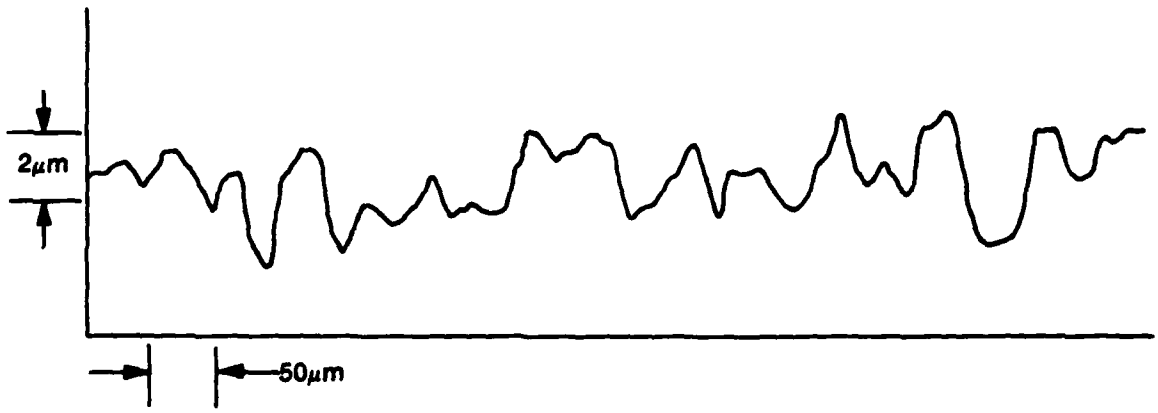



**Figure 4. Surface profile and Nomarski micrograph of a Si wafer taken before mechano-chemical polishing**

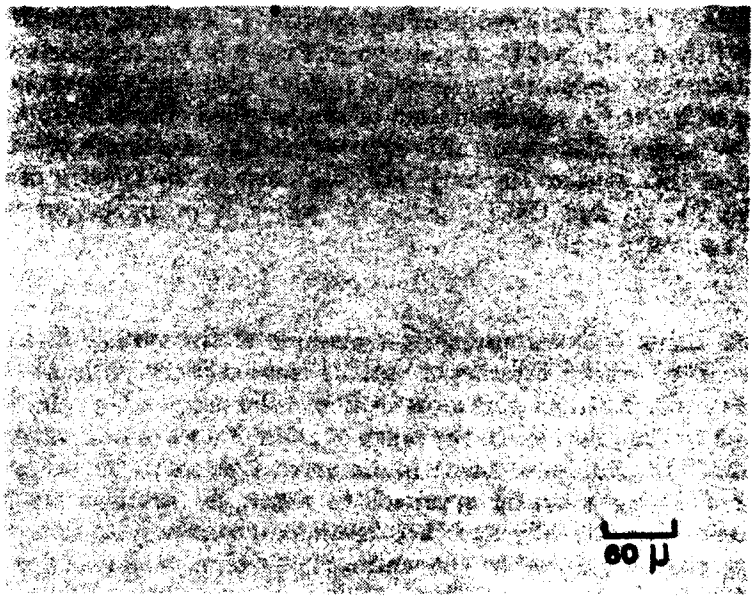




**Figure 5. Surface profile and Nomarski micrograph of a Si wafer taken after mechano-chemical polishing**

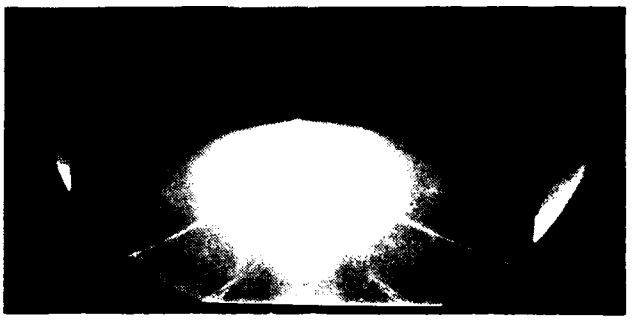


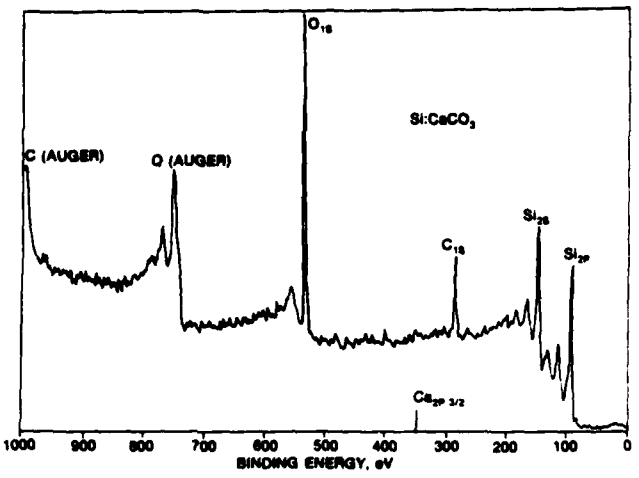
Figure 6. RHEED pattern of a mechano-chemically-polished silicon

ESCA spectra of a silicon sample wet mechano-chemically polished with $CaCO_3$ are shown in Figure 7. Spectrum (a) was obtained by scanning the polished surface, and spectrum (b) was obtained by scanning the sample again after sputter etching the polished surface to a depth of ~50Å. Features near $Si_{2P}$ peaks are shown on an expanded scale in (c). The corresponding spectra of a silicon sample wet mechano-chemically polished with $BaCO_3$ are shown in Figure 8. For comparison purposes, the spectrum obtained by scanning the fracture surface of silicon is shown in Figure 9. Note that there is no Ba or Ca contamination of the polished silicon surfaces, and that the peak at 103-eV almost disappears after sputter etching to a depth of ~50Å. The ratios of the height of this peak to the height of $Si_{2P}$ peak in the spectra of silicon surfaces polished with $BaCO_3$ and $CaCO_3$ is ~0.16, which is in agreement with the values reported by Yasunaga et al.[1]

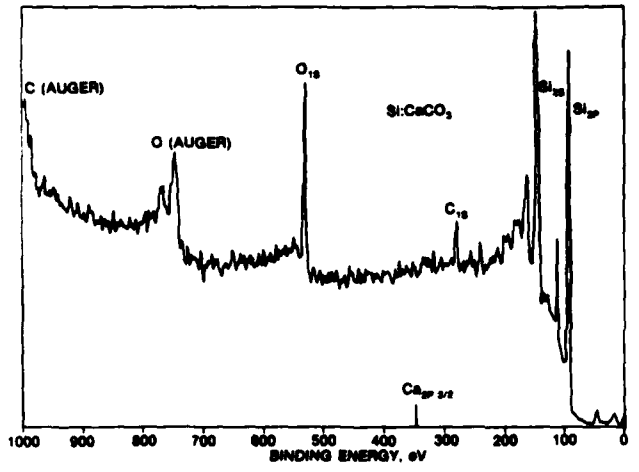
Measurements using an interferometric technique of the surface figures of mechano-chemically-polished silicon crystals of various cross sections indicate that the surface figure degrades with the increasing cross section of the sample. For samples of about 1cm × 1cm in cross section, flatness in the range of 1/5th of a wave to 1 wave (at 633nm) has been measured. For samples of 2.5cm in diameter, flatness in the range of 1λ to 3λ has been measured. Using a hybrid approach in which the samples are polished to the desired flatness conventionally and then mechano-chemically to remove the damaged layer and scratches introduced by conventional polishing, we have been able to achieve a flatness of 1/10 of a wave at 633nm. Figure 10 shows the surface figures of some mechano-chemically-polished silicon samples.

Several samples of mechano-chemically-polished silicon crystals were etched in a freshly prepared Wright etchant.[3] All these samples appeared scratch-free before etching. On the surfaces of some of these samples, a few isolated scratches appeared after etching for a period of about 1 minute (see Figure 11). It is believed that the chunks of silicon that often chip off from the periphery of the sample during mechano-chemical polishing are contaminating the lap and causing these scratches.

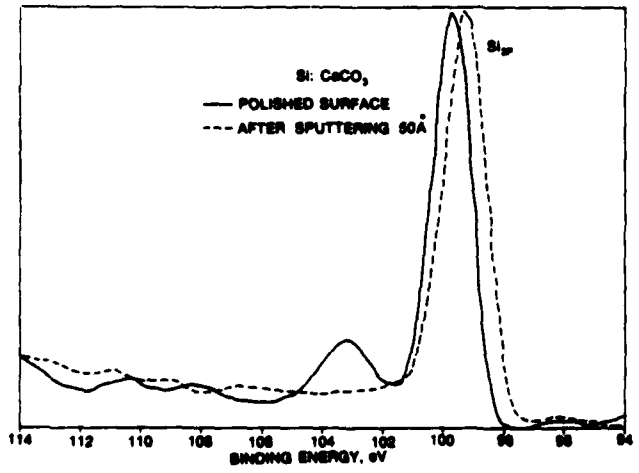
3. M.W. Jenkins, J. Electrochem. Soc., 124, 757, (1977).




(a) Polished surface

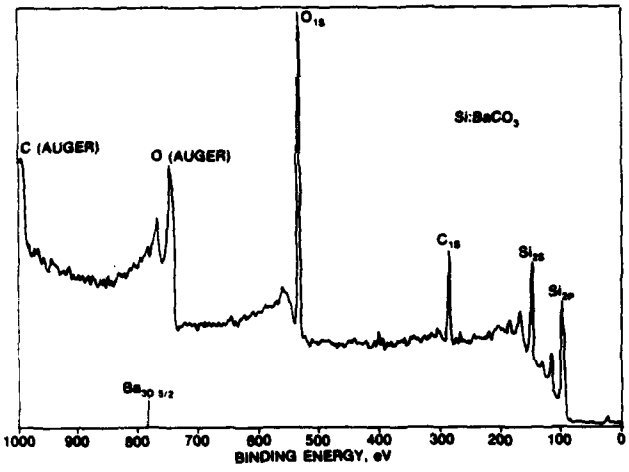



(b) After sputter etching 50Å from the polished surface

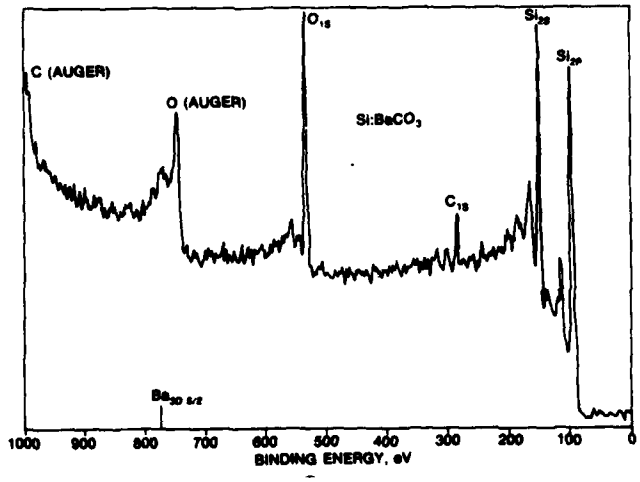



(c) Features near $Si_{2p}$ peak in spectra (a) and (b) shown on an extended scale

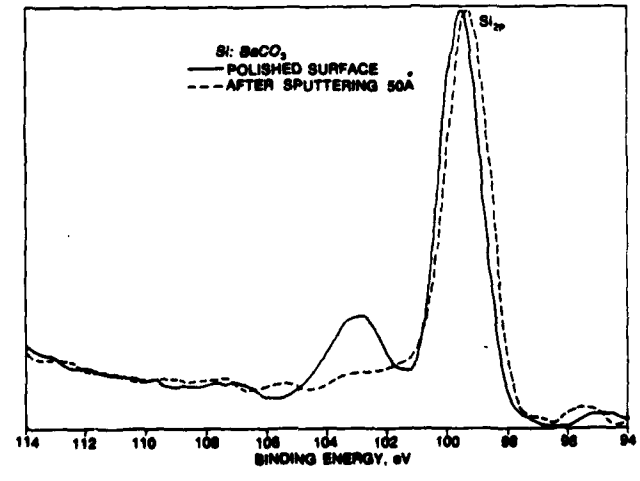
Figure 7. ESCA spectra of a silicon crystal polished with $CaCO_3$




(a) Polished surface




(b) After sputter etching 50Å from the polished surface




(c) Features near $Si_{2p}$ peak in spectra (a) and (b) shown on an extended scale

*Figure 8. ESCA spectra of a silicon crystal polished with $BaCO_3$*

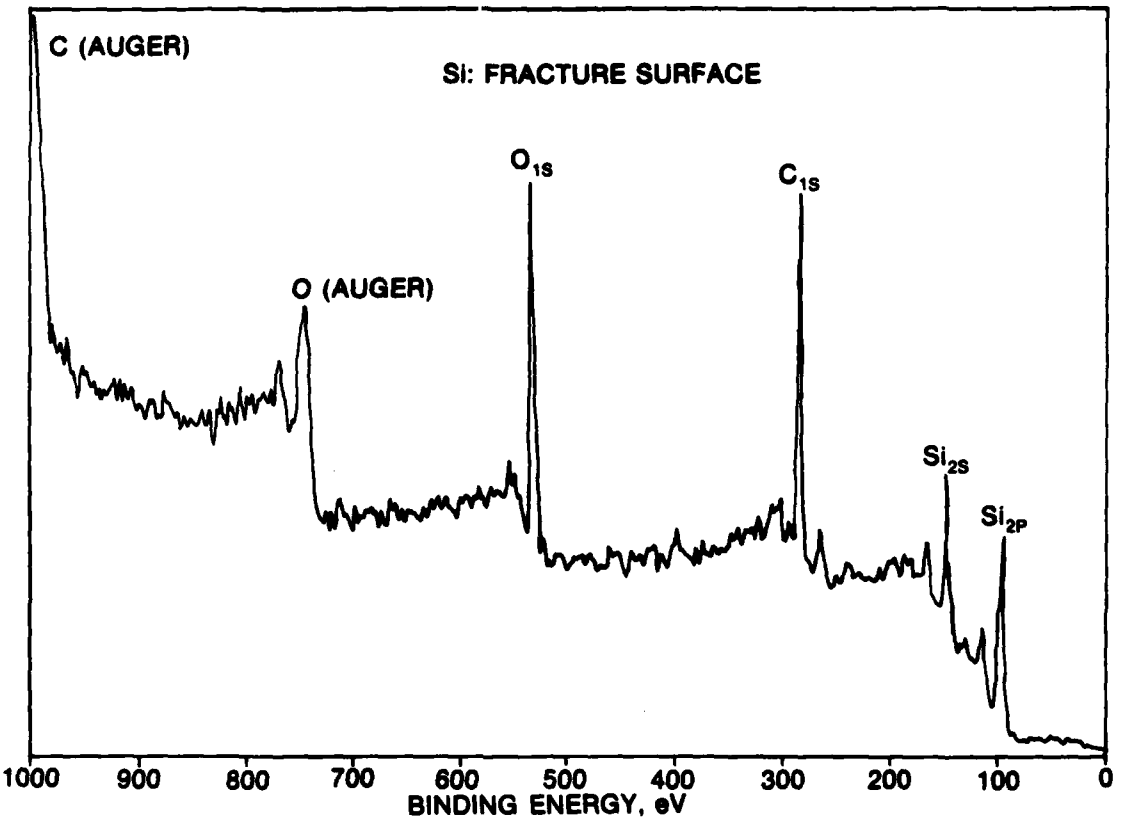
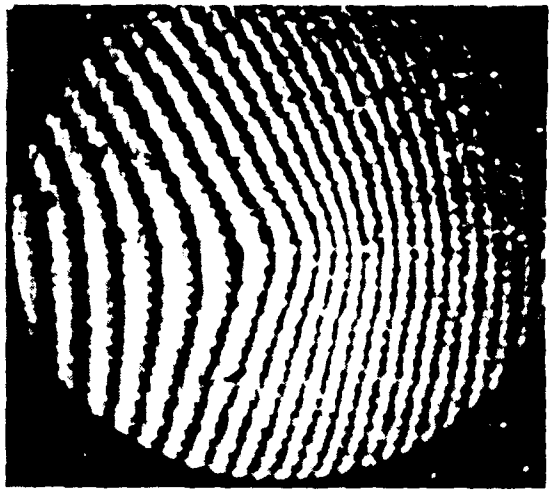
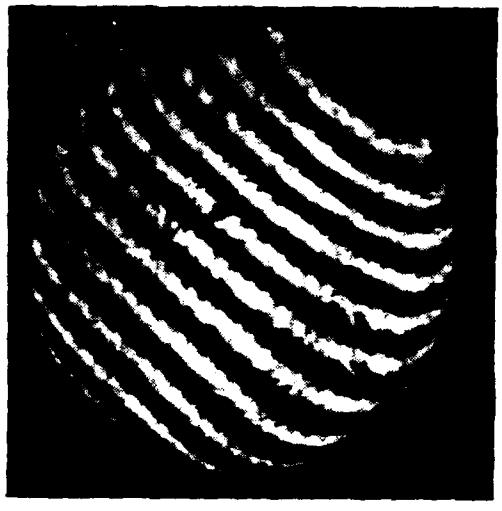



Figure 9. ESCA spectrum of fracture surface of silicon



(a) 3.2cm in diameter



(b) 2.54cm in diameter. Sample (b) was polished flat conventionally before mechano-chemical polishing.

Figure 10. Surface figures of mechano-chemically-polished silicon crystals

Figure 11. Scratches on the surface of a mechano-chemically-polished silicon made visible by Wright etch

## ALUMINA

All efforts to mechano-chemically polish alumina were made on the Strasbaugh Polishing Machine. Single-crystal alumina (sapphire) of $(10\bar{1}2)$ orientation and tape-cast alumina were starting materials. Tape-cast alumina was purchased from Coors and the sapphire from Kyocera.

Initial efforts to mechano-chemically polish $Al_2O_3$ were made on a lap of window glass using talc, NaCl, KCl, $Fe_2O_3$, and $SiO_2$ as abrasives. No significant mechano-chemical polishing of $Al_2O_3$ was observed when the glass lap was covered with talc, NaCl, or KCl. However, evidence for mechano-chemical polishing was readily obtained when $Fe_2O_3$ or $SiO_2$ was used as an abrasive. During this study, it was also observed that the presence of neither $Fe_2O_3$ nor $SiO_2$ was necessary to polish $Al_2O_3$. The contractor was able to obtain scratch-free samples of $Al_2O_3$ by simply polishing them on a plate of window glass without any abrasive. Most of the efforts were then concentrated on studying polishing of $Al_2O_3$ by window glass. Similar polishing effects were later observed when $Al_2O_3$ was polished on a lap of Pyrex glass.

To get an indication of the rate at which $Al_2O_3$ was being removed when polished on window glass, three tape-cast alumina samples of 1.25cm × 1.25cm in cross section were polished flat on a tin lap using 6-$\mu$m diamond paste. Several Knoop indentations were

then made on one of the samples using a load of 300g and all three samples were polished on window glass using a polishing load of 4.5kg and a lap rotation of 60 r/min. The decrease in lengths of long diagonals of Knoop indents were measured as a function of polishing time for a period of ~2 hours, the time it took to obtain scratch-free surfaces on all three samples. From these measurements, the average removal rate of alumina over this period was computed. Using this same procedure, the average removal rates of tape-cast alumina and sapphire when polished conventionally using 6-$\mu$m diamond paste were calculated. The results are summarized in Table 2. It can be seen that removal rates obtained in mechano-chemical polishing are comparable to those obtained in conventional polishing using 6-$\mu$ diamond.

Following the removal rate measurements, attempts were made to polish sapphire samples of 2.5cm × 2.5cm in cross section and tape-cast alumina samples of 5cm × 5cm in cross section. A considerable length of time was required to mechano-chemically polish these samples (e.g., ~2 days to simultaneously polish three sapphire samples and ~5 days to simultaneously polish three tape-cast alumina samples). To prevent the sample holder from getting too hot during these experiments, water was periodically sprayed on the lap. The polishing load used was of ~15kg during polishing of sapphire samples and ~25kg during polishing of tape-cast $Al_2O_3$. At higher loads, the glue used to mount the samples to the sample holder failed and excessive chipping occurred at the edges of the samples. Optical microscopic examinations during these experiments indicated that the removal rates were high in the beginning but decreased rapidly as greater and greater fractions of sample cross sections became smooth. These observations are similar to those made by Ikeda, et al.,[4] while mechano-chemically polishing sapphire on silicon laps.

**TABLE 2. POLISHING RATES FOR ALUMINA**

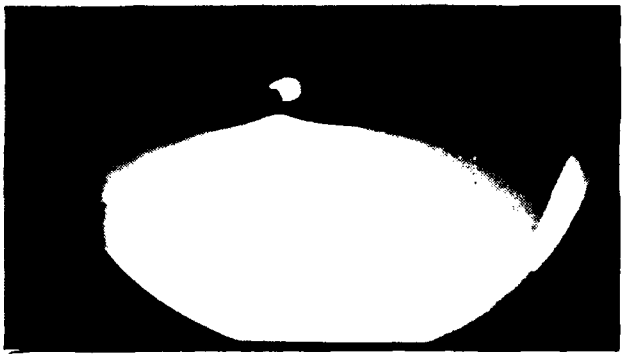
| Sample | Polishing Method | Polishing Rate |
|---|---|---|
| Sapphire ($10\bar{1}2$ face) | Conventional, 6-$\mu$m Diamond | 3.3Å/sec |
| Tape-cast Alumina | Conventional, 6-$\mu$m Diamond | 4.5Å/sec |
| Tape-cast Alumina | Mechano-chemical, Window Glass | 3.2Å/sec |

4. M. Ikeda, A. Yamada, Y. Kokaji and S. Kimura in The Science of Ceramic Machining and Surface Finishing II, B.J. Hockey and R.W. Rice, editors, National Bureau of Standards Special Publication 562, 1979, p. 562.

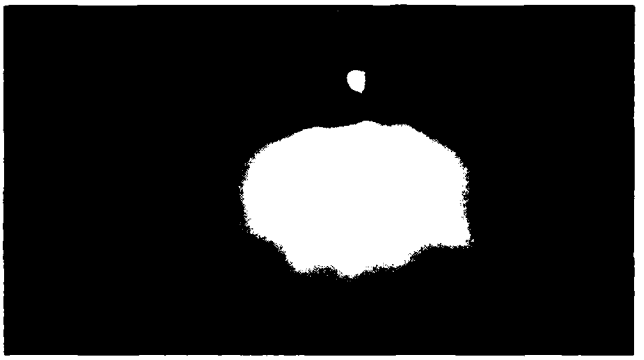
Figure 12 shows the RHEED patterns taken from a sample of Kyocera sapphire before and after mechano-chemically polishing on a window glass lap. Complete absence of any lines or spots in the diffraction pattern taken before mechano-chemical polishing revealed the presence of a thick damaged layer on the surface. Much of this damaged layer was removed by mechano-chemical polishing, as indicated by the presence of spots and Kikuchi lines in the diffraction pattern taken after mechano-chemical polishing.

Optical microscopic examinations of mechano-chemically-polished alumina samples revealed few scratches. Nomarski micrographs and surface profiles of polished surfaces are shown in Figures 13 and 14, respectively. It is seen that mechano-chemical polishing produces a much smoother surface on sapphire (peak-to-peak roughness of ~40Å) than on tape-cast alumina (peak-to-peak roughness of ~2500Å). The smoothness in the case of tape-cast alumina is perturbed by the presence of grain boundaries, second phases, and porosity.

Surface figures of several mechano-chemically-polished samples of alumina were measured using a Tropel Interferometer. As in the case of silicon polished on a linen Bakellite lap, the surface figures of mechano-chemically-polished alumina degraded with the increase in the polished surface area from a value of ~1/5th of a wave (at 633nm) for samples of 1.25cm × 1.25cm in cross section to a value of ~3 waves for samples of 5cm × 5cm in cross section. Surface figures of mechano-chemically-polished sapphire crystals are shown in Figure 15.

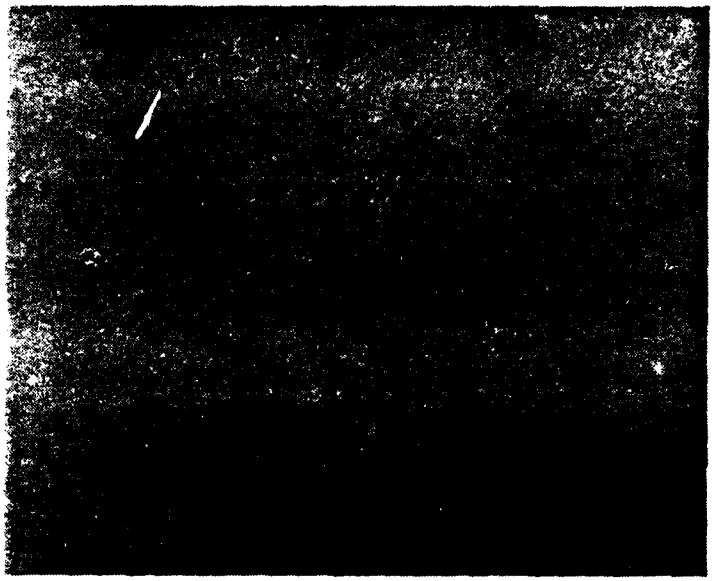


(a) Before mechano-chemical polishing



(b) After mechano-chemical polishing

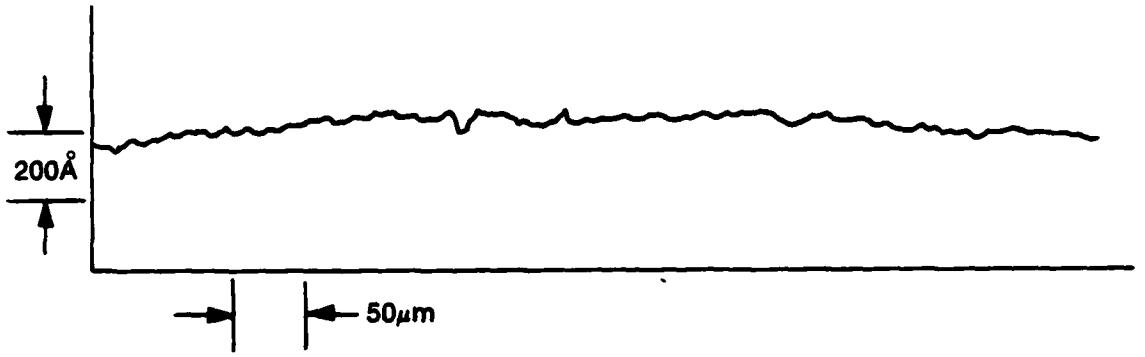
Figure 12. RHEED patterns of sapphire



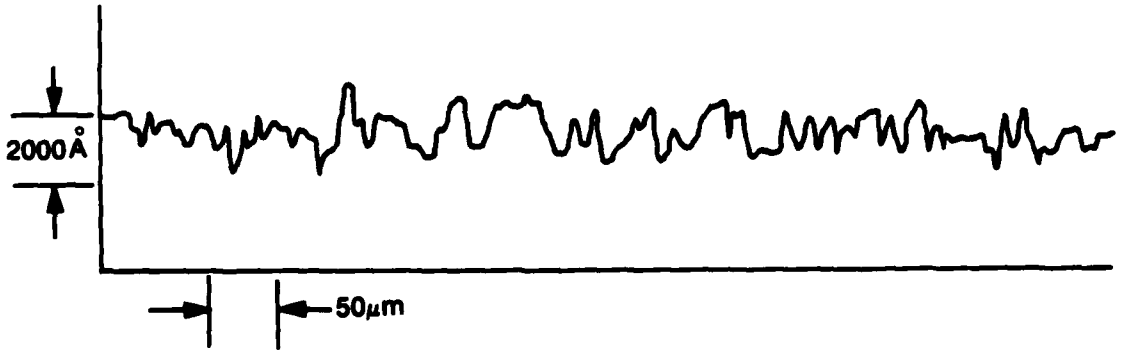
(a) Sapphire

(b) Tape-cast alumina

Figure 13. Nomarski micrographs of mechano-chemically-polished sapphire and tape-cast alumina

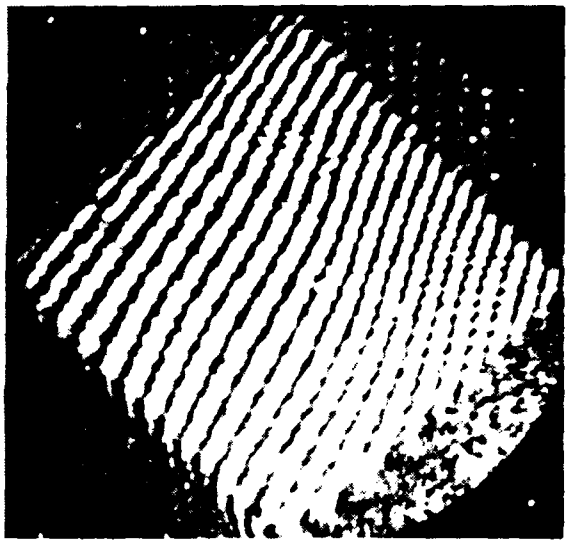



(a) Sapphire

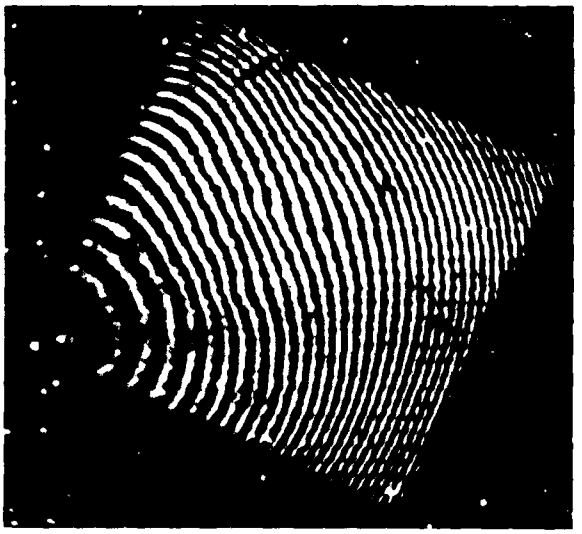



(b) Tape-cast alumina

Figure 14. Surface profiles of mechano-chemically-polished sapphire and tape-cast alumina



(a) Sample cross section
1.25cm × 1.25cm



(b) Sample cross section
2.5cm × 2.5cm

Figure 15. Surface figures of mechano-chemically-polished sapphire

# Section 4
# Discussion

Thus far, work performed in this program confirms the occurrence of mechano-chemical polishing effects in the case of Si and $Al_2O_3$. In addition to $CaCO_3$ and $BaCO_3$ abrasives reported by Yasunaga et al.,[1] MgO was identified in this work as another suitable soft abrasive for mechano-chemical polishing of silicon.

Much of the published work on mechano-chemical polishing refers to single-crystal samples of small cross section, typically 1cm × 1cm. Attempts were made in this program to apply this technique to single-crystal and polycrystalline samples of much larger cross section (silicon single crystals of up to 46cm$^2$ in cross section and alumina samples of up to 25cm$^2$ in cross section). It was observed that silicon samples of this size can be polished mechano-chemically in a period of hours, while considerably longer times (4-5 days) were required to polish $Al_2O_3$ samples. This difference in the behavior of silicon and alumina is not clearly understood and needs additional study.

Evidence for mechano-chemical polishing in the form of scratch-free surfaces has not been obtained in the case of magnesia and it is concluded that magnesia is too soft to withstand pressures required for it to interact chemically with soft abrasives.

In order to extend the application of mechano-chemical polishing techniques to other materials, efforts will be made in this program to mechano-chemically polish GaAs and $Si_3N_4$. The hardness of GaAs lies between that of silicon and magnesia and it is also a material of great importance in the electronics industry. $Si_3N_4$ can be made by tape casting and by hot pressing with thermal conductivity comparable to that of alumina, and with a coefficient of thermal expansion that matches that of silicon. Because of these properties, $Si_3N_4$ is considered a potential substrate material for very high speed integrated circuit (VHSIC) packaging. This application requires flat, scratch-free surfaces containing minimum pullouts. Since mechano-chemically-polished surfaces of $Si_3N_4$ are likely to meet these requirements, there is a need to identify suitable soft abrasives for $Si_3N_4$.

One of the general objectives of this program is to understand the mechanisms involved in mechano-chemical polishing of ceramic materials. To meet this objective, efforts will be concentrated on further characterizing the mechano-chemically-polished surfaces, and on determining the structure and chemistry of reaction products.

Another general objective of this program is to determine the effects of surface finishing on the physical performance of materials. Research on another program at our facility concerned specifically with the techniques for placing high resolution conductor metallization on ceramic substrates has compared the different surface finishing methods. Thus far, the adhesion of chromium-copper thin film metallization has shown

no sensitivity to the smoothness of the surface finish. The suggestion is that the chromium bonds chemically to the surface to provide the excellent adhesion. It is possible that the less reactive copper might show lower adhesion strengths with smoother surfaces. Further work needs to be performed to understand the mechanisms involved.

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